INFLUENCE OF STRESS CORROSION ON STRENGTH OF GLASS FIBERS

(Unclassified)

First Bi-Monthly Progress Report

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I. STATEMENT OF PURPOSE

This program aims to elucidate the chemical or physical processes which influence the measured strength of glass fibers after they have been formed from the melt. Specifically, it is concerned with the effects of stress corrosion reactions which involve water vapor from the atmosphere or whatever other sources are available to the reacting species in the glass. The principal experimental technique is a systematic investigation of static fatigue, or delayed failure of single virgin fibers in tension under various environmental conditions.

II. INTRODUCTION

In the course of recent studies in this laboratory sponsored by the Special Projects Office of the Navy Bureau of Weapons, a considerable body of information has been obtained which indicates that a stress corrosion reaction involving water vapor is a substantial factor in reducing the measured strength of glass fibers.[1]

It is generally assumed that even pristine fibers, taken directly from the bushing under carefully controlled conditions, still contain a certain population of flaws and that the most critical flaw in a particular fiber sample determines the instantaneous strength of that segment. Our previous experiments have shown that the measured strength at normal room temperature and humidity is much lower than that which the fibers exhibit at very low temperature. The difference is much greater than that which can be explained on the bases of a change in elastic modulus with temperature. For example, the median strength determined at -196°C was 825,000 psi, while at room temperature it was only 510,000 psi. This is a 60% higher strength at low

(1) See Final Report, High Strength Glass Fibers Development Program - Task 2, May 20, 1963, Contract NOw61-0641-c(FBM), by D.L. Hollinger, H.T. Plant and R.F. Mulvey.

temperature, whereas the change in Young's modulus for this type of glass is less than 5% over this temperature range. Considering all the evidence from other investigations as well as our own, we believe that a time, temperature and stress-dependent corrosion reaction is indicated and that the reactants involved include water vapor and alkali ions as well as the silica backbone of the glass structure.

Although such an explanation is readily deduced from the experimental information, there are numerous questions still unanswered. For instance, we need to know more about the degree to which the rate of the corrosion reaction is dependent on stress. It is also important to know whether there exists a lower limiting stress below which corrosion is not enhanced and does not lead to reduced strength. The effect of duration of a certain level of stress upon the fiber's ability to sustain a subsequent higher or lower load is another question yet to be answered.

Many of the above problems could be more clearly understood if sufficient data on static fatigue were available. It is to fill this need that the present program has been undertaken.

III. CURRENT PROGRAM

A. General Concepts

Single fibers of E-glass, with a nominal diameter of .0004-inch are the samples to be tested. These fibers, as in all our prior work, will be drawn in our own facility, from a single orifice, platinum crucible. A description of the furnace and associated fiber-drawing equipment may be found in the previously mentioned Final Report on the preceding Navy contract. Details of the procedure used in taking samples for test are also given in the referenced report.

For our new experiments on static fatigue, fixed tensile loads will be applied to the individual fibers and the time to fail recorded for various environmental conditions. It is anticipated that at normal room temperature and humidity, delayed failure will be recorded such that the time to fail will be some function of the applied load. This implies that failure is influenced by the rate of one or more processes. From the particular relationship which is revealed between the applied stress and the time to fail, it may be possible to deduce the

actual degree by which the governing processes are a function of the stress. Similar work with other materials has sometimes shown the existence of a fatigue limit, or stress level below which time to fail becomes immeasurably long.

The effect of temperature on the relationship between stress and time to fail will suggest the kinds of processes most likely involved in failure. It is already known from our prior work that the short term strength of fibers is considerably higher at liquid nitrogen temperature (-196 °C) than at room temperature. Therefore, the loads applied to fibers in low temperature static fatigue tests will generally be higher than those used at room temperature. In addition, it is anticipated that the effect of stress level on failure time should be much less, and possibly absent altogether at liquid nitrogen temperature. This would be the case if a stress-dependent chemical corrosion reaction is responsible for the lower strengths in normal environments.

To determine the contribution of an external reactant (i.e. atmospheric water vapor) to strength loss at room temperature, static fatigue tests will

also be conducted in the presence of an active desiccant. Our earlier work on testing of fibers in ether solutions of lithium aluminum hydride made some progress in this difficult task. In our present program we shall use a hydride solution from which the lithium ions have been removed by titration. It is hoped that the aluminum hydride remaining will still effectively remove surface moisture from the glass without the danger of modification of the structure of the glass itself by the mobile and reactive lithium ions.

B. Specific Program

The work statement for this contract is included here as it provides a clear resume' of the actual experiments to be performed.

"The Contractor shall conduct a study of the influence of stress corrosion on the strength of glass fibers. Said research shall include, but not necessarily be limited to, the following:

(1) E-glass fibers of approximately 0.0004-inch diameter shall be drawn from a single orifice crucible. These fibers shall be produced in normal atmospheric humidity, wound on collecting forks and

stored in closed containers over silica gel until ready for mounting and testing. Care shall be taken in handling fibers to insure that no solid object touches the portions to be tested, but normal laboratory atmosphere shall be maintained.

(2) Static fatigue tests shall be carried out at room temperature and normal humidity with dead weight loading applied within less than one second. Loads shall be chosen to give delayed failures covering at least (5) decades of time. At least twenty (20) tests shall be made at each of at least five (5) predetermined loads.

(3) Similar static fatigue tests shall be conducted at liquid nitrogen temperature. In this case the loads chosen shall be based upon fixed multiples of the median short term room temperature strength determined in Item (2), above. Short term strength shall be considered as the applied load which results in failure within five (5) seconds or less. It is anticipated that the multiples of such strength used in low temperature tests will be among the following: 0.8, 1.0, 1.2, 1.4, 1.6, and 1.8.

At least twenty (20) tests shall be made at each predetermined load. At least five (5) different load levels shall be chosen.

(4) Static fatigue tests shall also be carried out at room temperature in an active desiccant (e.g. aluminum hydride in ether solution or fuming sulfuric acid). As in Item (3), above, loads shall be chosen equal to multiples of the median room temperature short term strength in normal humidity. The factors in this case will have to be determined by trial, since there is not sufficient prior data on short term strength in desiccants.

Every reasonable effort shall be made to keep the glass dry in preparation for these tests. This shall entail preparing at least twenty (20) specimens by drawing through a tube of dry nitrogen or other gas near the bushing instead of in ambient air. If this does not alter the result of the tests in an active desiccant, then drawing in air as usual may be used in preparation for the tests of Item (4).

(5) The data shall be processed for determining statistical significance of the trends indicated. If the number of tests has not been sufficient, the analysis should indicate this.

(6) The results shall be examined in the light of existing physical-chemical rate theory for stress corrosion processes as developed by R.J. Charles and others. In particular, the investigators should try to determine if a single law describes or predicts the results over a wide range of stresses and if a safe range of stress exists in which the "Joffé affect" prevails. The possibility of finding a rule for integrating the loss in life shall be examined so as to predict the time to fail under varying stress as a function of time.

It should be kept in mind that the objective is to lay the groundwork for future improvement of the strength and strength retention of glass by commercially feasible processes and that complete understanding of how stress plus moisture affect the glass in detail is presumably useful to that end.

In explaining the experimental results and in formulating predictive laws or rules, the Contractor should draw on any other useful data in addition to that acquired during this contract. In particular, the test results on previous Contract NOw-0641-C FBM should be used wherever applicable."

IV. PROGRESS TO DATE

A. Fiber Drawing Equipment

Some minor repairs to the electric furnace for melting the glass have been completed and the unit checked out at operating temperature. All glass remaining in the platinum crucible from the previous program has been carefully removed. It was necessary to resort to digestion of the glass in boiling, concentrated sodium hydroxide solution for removal of the last traces of old glass which bonded tenaciously to the platinum. The crucible has since been refilled with E-glass marbles and the melt allowed to fine at 2400°F for about 6 hours. Fibers were drawn continuously at low drawing speeds with no difficulty, but were neither measured nor tested for tensile strength. The equipment is all performing satisfactorily.

B. Static Fatigue Test Equipment

A relatively simple setup for conducting static fatigue tests at either room temperature or liquid nitrogen temperature has been assembled. Essentially, we have a tank in which the fiber samples are enclosed and which provides the appropriate environment throughout

the test period. Each fiber is connected through a suspension train to a switching device which automatically activates the correct timers whenever a fiber is placed under load or whenever one breaks. There are 12 equivalent positions in this equipment and we plan to run duplicate tests to achieve a total of 24 replicas for each load point.

One important consideration in static fatigue testing of such tiny fibers is the elimination of all shock and vibration loads which could easily have a large effect on recorded breaking times. It is relatively simple to isolate the test equipment from moderate and high frequency vibration, but making it insensitive to jarring and low frequency oscillations is a more difficult problem. However, we were fortunate to have in the laboratory a solid concrete pedestal situated on a solid footing which could accommodate the test equipment. Measurements made on the test equipment after installation in this location, under the worse possible shock conditions likely to occur, showed a peak displacement amplitude of less than .000020-inch, as read on an IRD Vibration Analyzer.

C. Program Design and Presentation of Data

The experimental design, or program of tests, has been set up on a preliminary basis as shown in Table 1. It will be noted that for each value of load, there are two groups of samples designated A and B and that each of these groups consists of 12 individual fibers. This breakdown helps in the randomization of samples in order to minimize any possible uncontrolled variable such as sample history prior to test, etc. In order that the necessary elapsed time between testing the various groups of fibers will not constitute an important variable, we shall allow all samples an unstressed storage period, in equilibrium with Drierite, of at least one week prior to testing. It is currently planned that some preliminary fatigue testing at room temperature will be necessary to establish the range of loads which are adequate to give useful failure data over an appropriate time span.

From our previous results on short term strength testing at liquid nitrogen temperature, we are able to estimate the factors by which room temperature loads can be multiplied for low temperature testing.

Thus, we can expect that certain percentages of the fibers will fail almost instantaneously when loads are applied at liquid nitrogen temperature equal to 1.4, 1.6, 1.8, etc. times the median short term room temperature strength. In the absence of any fatigue phenomena, these percentages would be the only fibers to fail under the given loads regardless of the test duration.

For the tests in active desiccant at room temperature, there will be need again for some preliminary experimentation to determine a proper load range. Although the existing data are quite meager, we should expect that loads somewhat higher than those used for normal humidity, room temperature tests would be necessary.

All data will be initially plotted as percentage of fibers failed versus time, with load as a parameter. It is hoped that analyses of the information obtained under the various environments will help us to clarify and extend the theory of failure which applies to E-glass fibers.

V. FUTURE PLANS

During the next reporting period, we shall have the following specific objectives:

1. Conduct preliminary room temperature tests to determine load range for fatigue program.
2. Complete statistical design of experimental conditions and sample randomization.
3. Draw all fibers to be used in entire static fatigue program.
4. Carry out static fatigue program at room temperature and normal humidity.
5. From results of Item 4, choose loads to be applied at liquid nitrogen temperature.

1

PROGRAM DESIGN -

Load	I						II					
Group	A			B			A			B		
Environment	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z

Order of Test --------------------- Not fina

Notes:

1. Loads I - V will be chosen to yiel

2. Groups A and B each consist of 12
unstressed, in equilibrium with Dr

3. Environments:

X is normal room temperatures an

Y is -196°C in nitrogen gas.

Z is active desiccant.

TABLE 1

I - STATIC FATIGUE TESTS

	III						IV						V					
	A			B			A			B			A			B		
Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z	X	Y	Z
nally determined-----------------------------------																		

eld meaningful data for each environment.

2 individual fibers. All groups will be stored, Drierite for at least one week prior to test.

and humidity.